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VISIONS, CONCEPTS AND STRATEGIES FOR SMART NITINOL ACTUATORS AND COMPLEX NITINOL STRUCTURES PRODUCED BY ADDITIVE MANUFACTURING

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ABSTRACT

This work covers different aspects of additive manufacturing of Nitinol parts. Firstly, requirements for the powder material and guidelines for the powder preparation are described in detail because the use of proper powder is essential for additive processing of high quality parts. Next, this work presents examples for Nitinol actuators, smart structures and devices which are produced by additive manufacturing. By demonstrating the functionality of these parts (e.g. shape recovery behavior after deformation), this work clearly points out a high potential for additive manufacturing of Nitinol. Moreover, additive manufacturing might even be able to open up new perspectives for Nitinol devices that have yet to be imagined.

INTRODUCTION

Shape memory alloys (SMAs) have characteristics which are unique in comparison to alternative actuating principles. In general, the competitiveness of an actuating system depends on its quality and precision, simplification and robustness, its mass and required space and certainly on its costs. Especially for the latter reasons, SMAs are interesting for actuating systems because a shape memory actuator consists of fewer components than conventional ones. Hence, by using SMAs, the space and the mass of the actuating system can be reduced. Today, the most common SMA is Nitinol due to its outstanding functional characteristics and the considerable high working capacity compared to other SMAs and even compared to conventional smart materials and actuators.

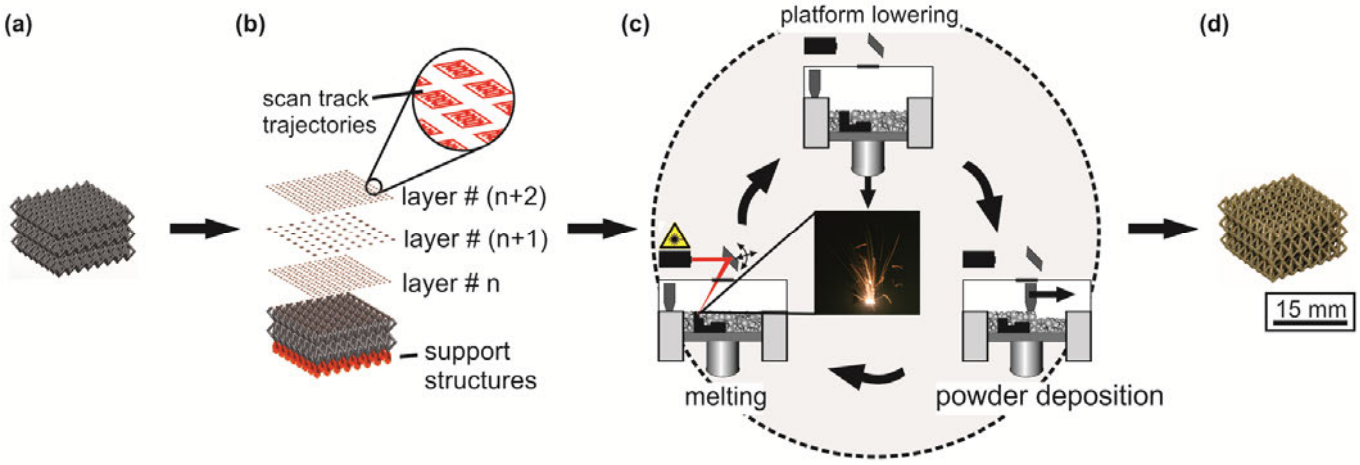

Figure 1. Scheme of the AM processing principle: CAD model (a), sliced CAD model prepared for AM (b), cyclic AM procedure: melting->platform lowering->powder deposition (c), complex Nitinol structure produced by AM.

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Nevertheless, processing and machining of Nitinol is by no means easy [1, 2]. The high elasticity and high ductility of Nitinol results in heavy tool wear during machining which makes the process difficult [3] and expensive, while grinding results in high heat generation which can affect the functional and structural properties of the part. For that reason most of Nitinol devices are made of simple geometries which are based on the same semi-finished products e.g. sheet, strip, wire, tube or bar. Hence, the potential of Nitinol cannot be fully exploited today due to that lack in manufacturing [4]. Additive manufacturing (AM) provides freeform technologies like selective laser melting (SLM) and, respectively, sintering (SLS) which is predicted to overcome the manufacturing difficulties. Those technologies allow for the direct production of parts from a sliced CAD model by laser-melting powder material in layers. Moreover, because of its layer-based processing principle AM can produce parts which cannot be produced by any other method. Figure 1 demonstrates the freeform of design given by the layer-based processing principle of AM.

However, for processing Nitinol, AM has not gained much attention so far. Furthermore, AM is not applicable for the production of every kind of part and AM will not substitute conventional processing routes at all. Figure 2 schematically demonstrates that AM provides an economic production route only for highly complex parts in small batches. Nevertheless, Worden et al. point out that Nitinol parts are regarded as being comparatively expensive especially because of small production batches or single devices [5]. In addition to the poor machinability of the material, AM clearly offers an attractive production route for highly complex Nitinol parts.

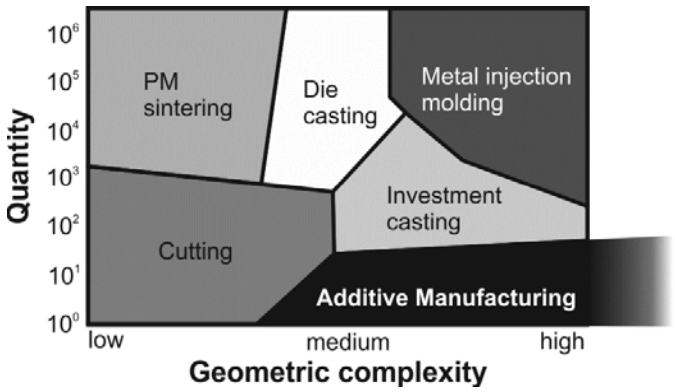

Figure 2. Economic comparison of different manufacturing techniques [6].

RAW MATERIALS

The choice of the proper powder material is essential for optimized AM processing. Nowadays, several powder materials (steel, Titanium, Ni-based high-temperature alloys, Cobalt-Chromium, etc.) are commercially available for AM but Nitinol powder is not qualified yet. Hence, one has to start from ingot material which has to be processed to powder (see Figure 3 (a) and (b)).

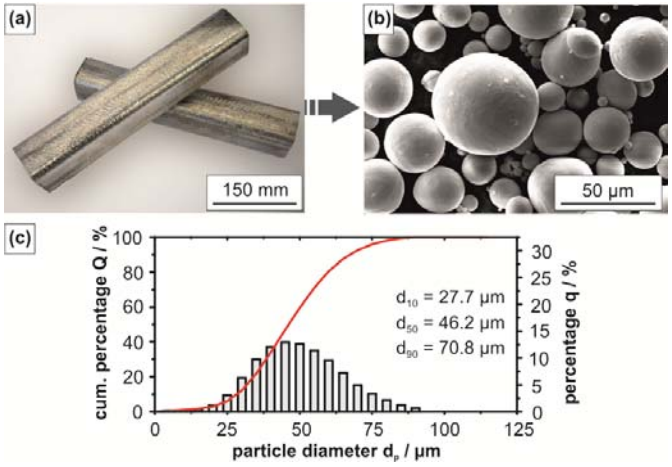

Figure 3. Nitinol ingots (a), Nitinol powder (25-75 µm) atomized by EIGA method (b), particle size distribution of this fraction (c).

In general, the powder particles should be as small as possible for the deposition of thin powder layers. The powder fraction has to have a multimodal particle size distribution (see Figure 3 (c)) for increasing the packing density [7, 8]. It is obvious that a high packing density of the powder bed will support the production of highly dense parts more than a very loose powder bed. For the use in AM, powders are usually gas atomized from ingot materials. Gas atomization allows for the production of spherical particles (see Figure 3 (b)). Spherical particles are required because they show a good flowability compared to sharp-shaped particles, which tend to hook at each other; hence the deposition of a thin and smooth powder layer may be impeded. Furthermore, the packing density of sharp-shaped particles might not that high because of the same reason. However, one has to consider that flowability and packing density of spherical particles both are affected by the particle size. Finer particles with a higher surface-volume-ratio lose their flowability due to increasing Van der Waals forces. Thus, they tend to adhere which significantly degrades the packing density, too. Previous studies showed that Nitinol particles in fractions smaller than 20-63 µm do not show flowability at all and even in the range of 20-63 µm the flowability is insufficient [9]. Those previous studies also considered the packing densities. Particle fractions of 25-75 µm and 45-100 µm show a packing density of about 60 % while a fraction of 20-63 µm exhibits a slightly reduced packing density of 56 %. Smaller particles show a significantly reduced packing density due to a reduced flowability. Fractions smaller than 45 µm exhibit a packing density of 44 % and fractions smaller than 25 µm even show packing densities of about 37 % [9].

Particle size, shape distribution flowability and packing density are general requirements for any powders used for AM.

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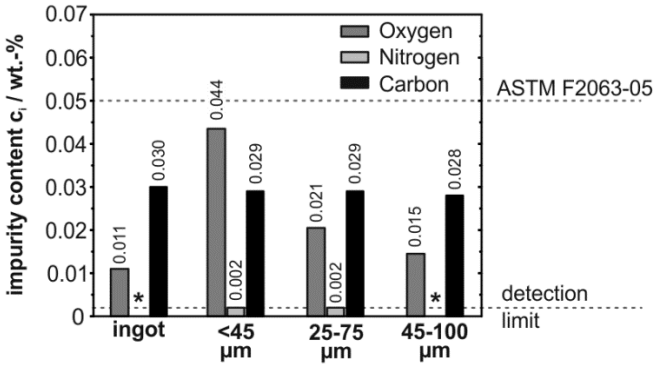

Figure 4. Impurity contents for several powder fractions prepared by EIGA.

Unfortunately, the preparation of proper Nitinol powder is more challenging because one has to consider additional requirements compared to the use of structural powder materials e.g. steel. It is well known that impurity related phases (e.g. oxides, carbides) strongly degrade the structural and functional properties of Nitinol [10-14]. Hence, Nitinol can only be atomized in an inert atmosphere to avoid high impurity pick-ups. It has been shown that electrode inert melting gas atomization (EIGA) is a very suitable process. This method eliminates the risk of impurity pick-up from a crucible because the molten material is contact-free [15]. However, even for this method, other authors [16] and previous studies [9] reported a significant increase of the Oxygen content with decreasing particle size due to a higher surface area (see Figure 4). One has to consider this, too, when choosing the most suitable powder fraction because there will be a further impurity pick-up during AM.

Certainly, when processing Nitinol the powder should show the ability for phase transformations. This ability can be degraded or even impeded by high impurity contents and a shift of the chemical composition away from the range which correlates with the proper phase transformation temperatures. However, all powder analyzed so far (ranging in the chemical composition from 49.7 at.-% to 50.8 at.-% Nickel; and ranging in particle sizes smaller than 25 µm up to 100 µm) showed unrestricted phase transformations. But one has to consider that the phase transformation temperatures are affected by AM. In general, the phase transformation temperatures slightly increase although there is an impurity pick-up. This increase is due to partial evaporation of Nickel [9, 17-21] and the precipitation of Ni-rich phases [9, 19, 20].

As aforementioned, Nitinol powders are currently customized for AM today. Hence, the preparation considering these strict requirements is extensive and therefore a quite expansive procedure. The suitable powder mass fraction after atomization should be as high as possible to avoid high waste. Unfortunately, the particle size cannot be set precisely with the EIGA method, so that after atomization the powder feedstock is first provided with a broad particle size distribution. Specifically, the mass fraction is very low in small particle fractions; for fractions less than 25 µm it is only 6-11 % of the

Table 1. Qualitative classification of different powder fractions of their suitability for AM.

	<25µm	<45µm	20-63µm	25-75µm	45-100µm
particle size	●	◕	◑	◕	◕
spherical shape	●	●	●	●	●
flowability	○	○	◕	◕	◕
packing density	◑	◑	◑	◑	◑
impurity content	◕	◕	◑	◑	◕
transformation ability	●	●	●	●	●
mass fraction	◔	◕	◑	◑	◑

● = fulfilled requirement ○ = unfilled requirement

entire powder fraction (less than 45 µm: about 23 %, 20-63 µm: about 40 % and 45-100 µm: about 47 %). For medium-sized particle fractions between 25-75 microns, the mass fraction increases to more than 62 %.

Table 1 summarizes the considerations for choosing a suitable powder. It is proposed that medium-sized fractions between 25-75 µm are particularly suitable because this fraction is considered the best compromise of size, shape, flowability, packing density, impurity contents, transformation ability and mass fraction after atomization.

FUNCTIONALITY OF AM NITINOL PARTS

Shape Recovery

Understandably, the most important characteristic of Nitinol is the recovery of its prior shape after a supposed plastic deformation. This shape recovery is done either directly after unloading (pseudoelasticity or mechanical memory) or by heating up the material to the high temperature phase (pseudoplasticity or thermo-mechanical memory). However, these functional properties strongly depend on the microstructure of the material. All types of microstructural defects (e.g. dislocations, precipitates or secondary phases and impurity related phases) strongly affect the elementary processes of the martensitic phase transformation which is fundamental for the shape recovery. Hence, it is not a given that a material which was produced from a powder by melting with a laser will possess these functional properties.

However, in previous publications it is reported by us [9, 17-20] as well as by other authors [22-26] that Nitinol produced by additive manufacturing can show shape recovery after pseudoplastic deformation. To demonstrate these pseudoplastic properties, Figure 5 shows a simple actuator element fabricated from a Ti-rich alloy (49.7% at.-% Ni). This actuator element was tested in a special actuator test rig. Details on the set-up of this test rig are described elsewhere [27, 28]. In this experiment a meandering actuator (initial length: l_0 = 41 mm) is constantly loaded by 2.5 N. Due to this load the actuator is elongated up to

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l_1 = 102.5 mm in its low temperature phase (situation 1). Hence, this elongation of 61.5 mm corresponds to the theoretical maximum actuator stroke. Nevertheless, this elongation not only consists of pseudoplastic deformation but also of pure elastic deformation. Those elastic deformations are not affected by the phase transformation. In a next step, the actuator was electrically activated (7 A) and heated up for 15 s which results in the transformation into the high temperature phase. This results in shape recovery of the pseudoplastic deformation (situation 2). During cooling down the actuator was again elongated by the constant load. This procedure was repeated 15 times in order to determine the functional fatigue of the actuator. Figure 5 (c) shows the corresponding time-stroke-diagram of this experiment (heating phases are shown in gray). In the first cycle, the reversible deformation was almost 72% of the theoretical stroke but decreases in the following cycles. The decrease is the highest in the first cycles. This decrease reaches an almost constant level and is barely noticeable in later cycles. The stroke in the fifteenth cycle still about 58 % of the theoretical stroke. It is worth noting again that any elastic deformation caused by the constant load is not affected by the shape memory effect.

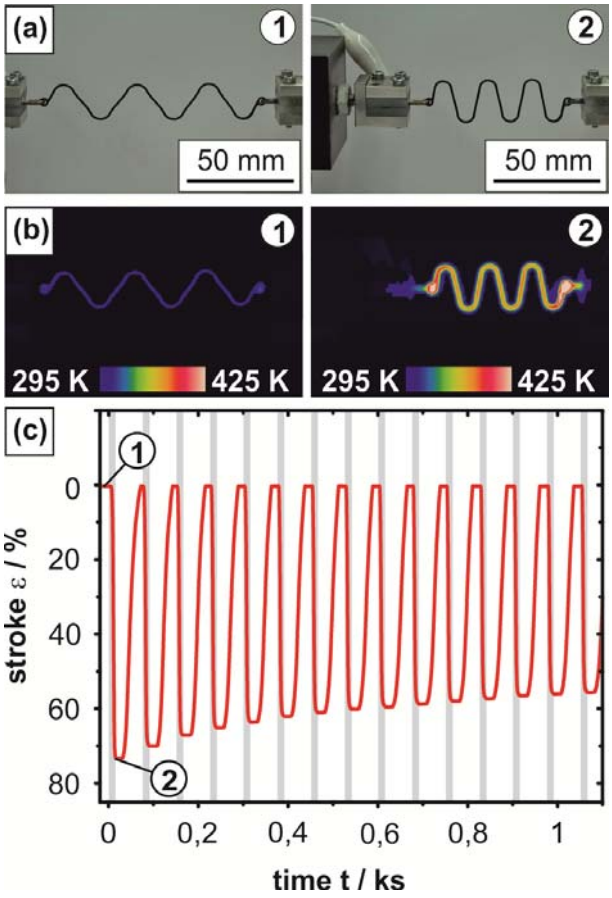

Figure 5. Photographs (a) and thermal images (b) of a Nitinol actuator showing a shape memory effect; corresponding elongation plot (c) [9].

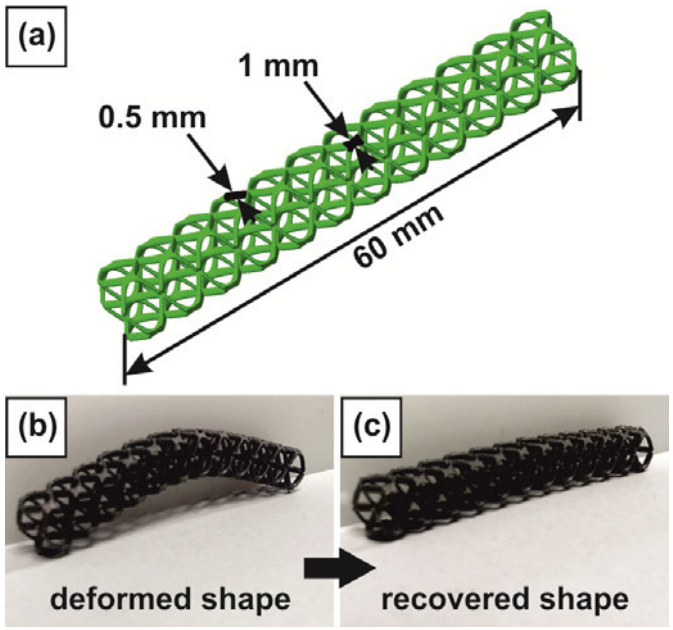

Figure 6. Complex Nitinol truss structure with filigree struts produced by AM: CAD model (a), structure performing the shape memory effect (b & c) [29].

Figure 6 shows another example for an actuator. While the meandering actuator presented in Figure 5 could be processed by conventional fabrication methods (e.g. forming from a wire), this complex truss structure is nearly impossible to fabricate by conventional methods. This filigree device (minimal strut diameter: 500 µm) is supposed to operate in a one-way shape memory regime. The structure gives the flexibility of having very light weight actuator with desirable stiffness and higher forces [29].

By choosing a proper alloy composition, additively manufactured Nitinol components show pseudoplastic properties directly after processing. Unfortunately, this does not apply to pseudoelastic applications. We showed in previous publications [9, 19] that subsequent heat treatments (solution annealing and aging) are required to establish pseudoelasticity. Halani et al. [30] recently published similar results but for another AM technique. It is well known for conventionally processed Nitinol that heat treatments, aging treatments in particular, are used to generate a specific state of precipitates in the material which supports pseudoelasticity [31, 32]. The formation of fine Ni-rich precipitates of type Ni_4Ti_3 strongly impedes dislocation plasticity by precipitation hardening and thus irreversible effects during deformation are reduced. Furthermore, fine Ni_4Ti_3 particles which are coherently dispersed in the Nitinol matrix support martensitic nucleation and thus pseudoelasticity is enhanced, too [33]. Figure 7 (a) shows a compression-unloading cycle of a dense monolithic Nitinol sample (50.7% at.-% Ni) which after AM processing was firstly solution annealed (950°C, 5.5 h, H_2O) followed by aging (350°C, 24 h, H_2O). Details on these heat treatments are

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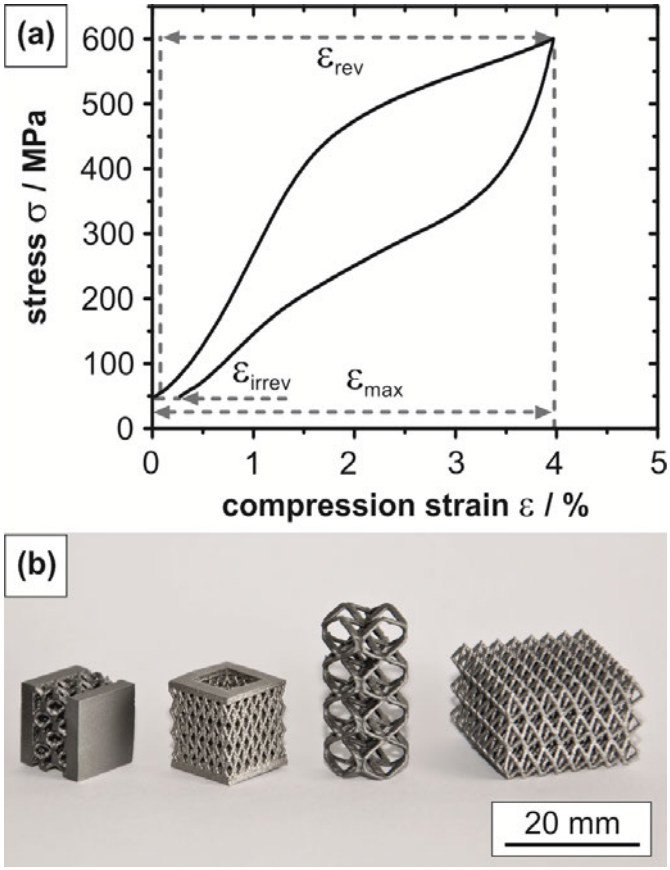

Figure 7. Stress-strain plot of a heat treated, dense AM Nitinol sample showing pseudoelasticity (a); complex AM Nitinol for future pseudoelastic applications [9].

given elsewhere [9, 19]. The material shows a stress-strain hysteresis between loading and unloading which is typical for pseudoelastic Nitinol and which accounts for high reversible strains. This sample was loaded up to 600 MPa which results in a maximum strain of about 4%. After unloading, only 0.2% of this deformation remains as irreversible strain while 3.8% is directly recovered. Future studies will analyze pseudoelasticity in highly complex porous structures like these presented in Figure 7 (b). However, it is noted at this point that heat treatments of those structures are by no means easy due to the large surface of the parts and the high reactivity of Titanium.

Medical Applications

Today, medical applications represent the most dominant market for Nickel-Titanium devices. Nitinol's outstanding functional properties make this alloy a prominent material for minimal invasive instruments (e.g. clot retriever), vascular-medical implants (e.g. self-expanding stents or filters), trauma surgery devices (e.g. osteosynthesis clamps) or for dental devices (e.g. orthodontic wires) [34-36]. In addition, Nitinol is of interest as a bone substitute material for load-bearing long-term implants due to its mechanical properties, although this application has not received much practice so far because of issues with processing. Furthemore, the alloy was initially stigmatized in the medical industry due to its nickel content, which is toxic in the human body.

In general, bone substitute materials are required to have damage tolerance and to have mechanical properties similar to the human bone. Nitinol demonstrates good deformability that is associated with the pseudoelastic behavior. This provides a damage tolerance, which is not demonstrated by any other metals or ceramics. Furthermore, stiffness of Nitinol (28-41 GPa for austenite) is very close to the stiffness of human bone (~ 1 GPa for cancellous bone, ~ 20 GPa for cortical bone). Common implant materials are much stiffer with Young's moduli greater than 100 GPa (Ti: ~110 GPa, Co-based alloys: ~193 GPa, stainless steel: ~ 210 GPa). This large stiffness difference between the implant material and the surrounding bone leads to an inhomogeneous stress distribution on the bone. This stress-shielding effect may cause bone resorption and weakens bone locally, which could result in aseptic implant loosening.

Nitinol shows, despite the high Nickel content, good biocompatibility, roughly comparable to that of Titanium or stainless steel. Basically, it is known that Nickel ions can cause allergic reactions. In high concentrations, Nickel can even be toxic and carcinogenic. The biocompatibility of Nitinol, however, is due to its strong corrosion resistance and the protective oxide layer. This TiO_2 layer is thermodynamically stable and has a low solubility in contact with body fluid. It minimizes Nickel-leaching out of the NiTi matrix in the body and protecs the surrounding tissues from direct Nickel contact.

As aforementioned, production of NiTi parts as bone implants is challenging due to poor machinability of the alloy. AM offers an attractive method for creating these implants. One main feature of this process is the possibility of creating implants from CT data of each patient. This allows for the production of tailored, patient-specific implants [37-39]. Furthermore, by using AM, implant materials can be designed with defined porous structures and surfaces with interconnected pores. This engineered porosity can allow ingrowth of both the existing bone (osseointegration) and blood vessels (vascularization) which can improve stable bone contact and reduce healing time after surgery.

Using both, the mechanical properties of Nitinol as well as the possibilities given by AM, Figures 8 (a) and (b) present another interesting application. Please note that the jaw segments in Figure 8 (a) are not manufactured from Nitinol but from stainless steel (316L, 1.4404). However, both jaw segments are additively manufactured using patient-specific CT data for preparing the CAD model. The Nitinol application shown in both figures is a root for a tooth which can be used when replacing a natural tooth. On the lower side, which is inserted in the patient's jaw bone, the root is designed with interconnecting pores for allowing osseointegration and vascularization. On the upper side this root has a plug for the patient-specific crone. These crones are typically manufactured from ceramics by conventional forming and sintering methods.

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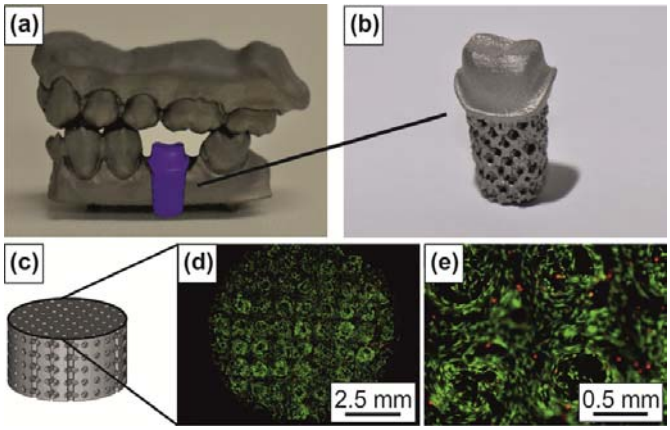

Figure 8. Example for an additively manufactured Nitinol medical application: schematic of a CAD model of a tooth root inserted in a lower jaw segment (a) and physical porous Nitinol root (b). CAD model of a porous specimen for analysis of biocompatibility (c), live-dead staining of AM Nitinol samples cultured with stem cells (d) and (e) [40, 41].

In recent years, the use of AM gained importance for producing crones, too. When applying AM these crones are additively manufactured from Cobalt-Chromium and subsequently coated with a ceramic. Figures 8 (c) and (d) show excerpts from a study our team has published with collaborators from biological and medical science. The purpose of this study was to assess the biocompatibility Nitinol processed when fabricated by AM [40, 41]. Samples varying in surface structure and porosity were cultured with human mesenchymal stem cells (hMSCs) and incubated for 8 days. We used a two-color, live-dead staining by fluorescence microscopy to analyze adherence and viability of the cells applied on our material. Using this method, living stem cells appear green while non-living stem cells appear red. We found a huge majority of living stem cells on our samples and only a few dead ones. We could not detect any toxic reaction or activation of the stem cells at all. Hence, we suppose a good biocompatibility of additively manufactured Nitinol. Nevertheless, this is only an excerpt from a very first study. Future studies and additional testing methods are required to prove the biocompatibility of AM Nitinol beyond a reasonable doubt. However, these first results are very promising for a production of Nitinol implants using AM.

CONCLUSIONS

Additive manufacturing offers an attractive method for the production of Nitinol shape memory or pseudoelastic parts and medical devices. While conventional powder metallurgical technologies are already established, additive manufacturing of Nitinol has only received little attention so far. This is even more remarkable since additive manufacturing can offer considerable flexibility in terms of part's geometry and complexity due to a layer based processing principle. Hence, this work aims to exhibit the suitability of additive manufacturing Nitinol alloys by demonstrating the functionality of additively processed Nitinol parts. In addition, requirements on the powder material are described in detail because the use of proper powder is essential, especially when processing sensitive Nitinol.

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